U-63-2

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TRANSLATION

OBTAINMENT AND PROPERTIES OF MONOCRYSTALS OF THE HIGH-MELTING METALS, TUNGSTEN, RHENIUM, TANTALUM, MOLYBDENUM AND NIOBIUM

By

Ye. M. Savitskiy, Ch. V. Kopetskiy, et. al.

FOREIGN TECHNOLOGY DIVISION

AIR FORCE SYSTEMS COMMAND

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FTD-TT- 63-270/1+2

UNEDITED ROUGH DRAFT TRANSLATION

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English Pages: 5

SOURCE: Russian Book, Issledovaniya Po Zharprochnym Splavam, AN SSSR Institut Metallurgii, Moskva, (Trudy), Vol. 9, 1962, pp 192-194

S/569-62-9-0

FTD-TT- 63-270/1+2

Date 6 May 19 63

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OBTAINMENT AND PROPERTIES OF MONOCRYSTALS OF THE HIGH-MELTING METALS, TUNGSTEN, RHENIUM, TANTALUM, MOLYBDENUM, AND NIOBIUM

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Ye. M. Savitskiy, Ch. V. Kopetskiy, A. I. Pekarev, and M. I. Novosadov

The electron-beam fusing of high-melting metals has great advantages over other methods of fusing, arc method, induction method, etc. The possibility of doing the fusing in a high vacuum, the facility and convenience in controlling the electron beam, the possibility of overheating by several hundred degrees above the fusion temperature determined this method heating for the zonal purification of the metals with the highest melting point, niobium, molybdenum, tantalum, rhenium, and tungsten.

For the zonal electron-beam fusion of metals in the Laboratory of Rare Metals and Alloys of the Institute of Metallurgy, named for A. A. Baykov, there was designed and assembled the laboratory device (Fig. 1) which enables one to purify metals and obtain monocrystals. The basic parts of the unit are a plate with a cantilever for fastening the specimen, a lead screw with a bracket for fastening and shifting the cathode, a glass hood, a mechanism for shifting the cathode, a vacuum system, and power sources for the cathode and anode electrical circuits. The vacuum system includes a prevacuum mechanical pump of the type VN-2 and a standard vacuum unit VN-05-1, consisting of a diffusion pump of the type N5, a slide, and a chamber with ionization and thermocouple vacuum meters. The vacuum system enables one to obtain a vacuum up to $3 \cdot 10^{-6}$ mm on the mercury column with a rate of pumping of 3,000 l/min. From the prevacuum pump into the working chamber a separate sleeve is inserted which enables one to do preliminary pumping with a warmed-up diffusion pump. There is provided also a carry-off to be connected up in the case of the need for a helium leak detector.

OBTAINMENT AND PROPERTIES OF MONOCRYSTALS OF THE HIGH-MELTING METALS, TUNGSTEN, RHENIUM, TANTALUM, MOLYBDENUM, AND NIOBIUM

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The electron-beam fusing of high-melting metals has great advantages over other methods of fusing, arc method, induction method, etc. The possibility of doing the fusing in a high vacuum, the facility and convenience in controlling the electron beam, the possibility of overheating by several hundred degrees above the fusion temperature determined this method heating for the zonal purification of the metals with the highest melting point, niobium, molybdenum, tantalum, rhenium, and tungsten.

For the zonal electron-beam fusion of metals in the Laboratory of Rare Metals and Alloys of the Institute of Metallurgy, named for A. A. Baykov, there was designed and assembled the laboratory device (Fig. 1) which enables one to purify metals and obtain monocrystals. The basic parts of the unit are a plate with a cantilever for fastening the specimen, a lead screw with a bracket for fastening and shifting the cathode, a glass hood, a mechanism for shifting the cathode, a vacuum system, and power sources for the cathode and anode electrical circuits. The vacuum system includes a prevacuum mechanical pump of the type VN-2 and a standard vacuum unit VN-05-1, consisting of a diffusion pump of the type N5, a slide, and a chamber with ionization and thermocouple vacuum meters. The vacuum system enables one to obtain a vacuum up to $3 \cdot 10^{-6}$ mm on the mercury column with a rate of pumping of 3,000 l/min. From the prevacuum pump into the working chamber a separate sleeve is inserted which enables one to do preliminary pumping with a warmed-up diffusion pump. There is provided also a carry-off to be connected up in the case of the need for a helium leak detector.

The specimen in the form of a little rod of the diameter of 3 to 5 mm is fastened vertically in molybdenom jaws with tantalum springs. With the heating up and expansion the springs permit the specimen to move freely in the vertical direction. The bracket with the fastened specimen is insulated from the plate. It serves as anode. A loop-shaped cathode made of tungsten wire of the diameter of 0.7 to 1.0 mm, or of tantalum foil of the thickness of 0.1 mm and width of 2 mm is fastened in steel holder. The current for heating the cathode is supplied to the holders with the aid of copper flexible bars. The bracket with the holders of the cathode shifts vertically along a lead screw, which extends from the working chamber through a conical vacuum connection. One of the holders of the cathode, together with the plate, is grounded. The focusing of the electrons emitted from the cathode is accomplished by two parallel molybdenom plates with an opening of the diameter of 5 to 7 mm. The distance between the plates is about 5 mm.

The lead screw is made to turn by an electric motor through a worm reducer and a belt transmission with a general transmission ratio of 1:1000. The regulation of the speed is accomplished by means of a change in the number of revolutions of the electric motor. This makes it possible to change the sheed of the shifting of the cathode relative to the specimen within the limits of from 8 to 60 cm/hr. Terminal switches projected from an extension of the lead screw stop the motor at the distance of the focusing plates from the specimen clamps, equal to 1 to 1.5 cm.

A rectifier consisting of stepup transformers and four kenotrons of the type KR-150 (KP-150) assures single-semiperiod rectification with a voltage equal to 3.6 kv and a maximum current of about 550 ma. The regulation of the working current and voltage is accomplished with the aid of smooth change in the voltage of the primary winding of the transformers within the limits of

from 0 to 220 v. The heating up of the cathode is regulated separately. The scheme proposed by us for power supply assures the maintaining and regulating of the temperature in the fusion zone in the mode of operation selected and eliminates overloads.

See page 3a for figure 1.

Fig. 1. Electric circuit of unit for purifying metals and obtaining monocrystals:
1--anode, 2--cathode, 3--focusing plates, 4--rectifier, T_1--transformer 220/3500, T_2T_3--heater transformers 220/12, 220/10, T_4--autotransformer

For the visual observation of the course of the fusion outside of the glass hood there is set up an objective through onto a screen there are projected in enlarged form the cathode heated up to 2,000--2,500°, focusing screens, and the zone of fused metal.

Before the zonal fusion the vacuum tempering of the metal rod is effected for its degasification. For this purpose the rod is heated up by electron bombardment over the whole length at a temperature by 100 to 300° lower than the fusion temperature.

The movement of the fused zone for purifying the metal and obtaining monocrystals was effected at the rate of 8 to 10 cm/hr. The mode of operation in the fusion for the metals investigated is shown in the table.

The rods obtained after the fusion were etched and one each of them there clearly appeared the faces of crystals. The hardness on the Vickers scale measured on transverse sections of monocrystals of the different metals had the following values: Mo - 170, Nb - 79, Ta - 75, Re - 154, W - 345 kg/cm².

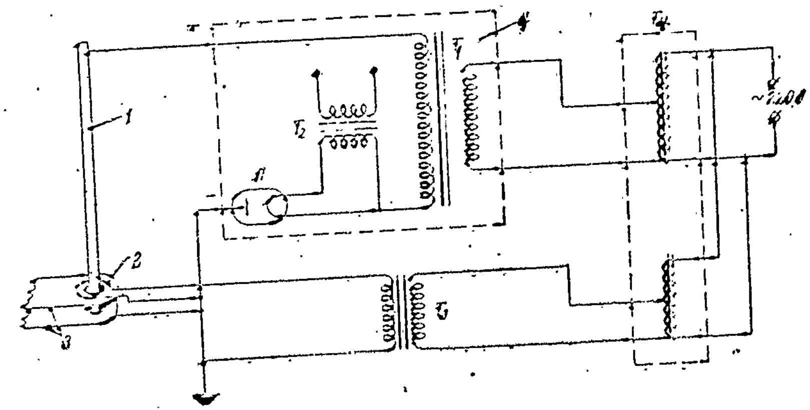

Figure 1.

Quality tests were also conducted of the bending and extension of monocrystals of tungsten and molybdenum. The extension tests of monocrystals of molybdenum, niobium, and tantalum showed cross-section contraction of about 100%

Metal	Diameter of rod, mm	Voltage v	Current ma
Nb	4	1200	110
Mo	4	1500	350
Ta	2	1800	150
Re	2,5	2300	220
W	4	2000	420

The obtaining of monocrystals of high-melting metals on the apparatus described above opens up new possibilities for the study of the properties of pure metals and their applications in various areas of new technology.

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